

## PATENT COOPERATION TREATY

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## INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY

(Chapter II of the Patent Cooperation Treaty)

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference */	<b>FOR FURTHER ACTION</b>		See Form PCT/IPEA/416
International application No. PCT/IT2004/000328	International filing date (day/month/year) 07.06.2004	Priority date (day/month/year) 30.07.2003	
International Patent Classification (IPC) or national classification and IPC C22B13/02, C01G21/02, H01M4/57			
Applicant CAM, S.R.L.			
<p>1. This report is the international preliminary examination report, established by this International Preliminary Examining Authority under Article 35 and transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p>3. This report is also accompanied by ANNEXES, comprising:</p> <p>a. <input checked="" type="checkbox"/> sent to the applicant and to the International Bureau a total of 4 sheets, as follows:</p> <p><input type="checkbox"/> sheets of the description, claims and/or drawings which have been amended and are the basis of this report and/or sheets containing rectifications authorized by this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions).</p> <p><input type="checkbox"/> sheets which supersede earlier sheets, but which this Authority considers contain an amendment that goes beyond the disclosure in the international application as filed, as indicated in item 4 of Box No. I and the Supplemental Box.</p> <p>b. <input type="checkbox"/> (sent to the International Bureau only) a total of (indicate type and number of electronic carrier(s)) , containing : sequence listing and/or tables related thereto, in computer readable form only, as indicated in the Supplemental Box Relating to Sequence Listing (see Section 802 of the Administrative Instructions).</p>			
<p>4. This report contains indications relating to the following items:</p> <p><input checked="" type="checkbox"/> Box No. I Basis of the opinion</p> <p><input type="checkbox"/> Box No. II Priority</p> <p><input type="checkbox"/> Box No. III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p><input type="checkbox"/> Box No. IV Lack of unity of invention</p> <p><input checked="" type="checkbox"/> Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p><input type="checkbox"/> Box No. VI Certain documents cited</p> <p><input type="checkbox"/> Box No. VII Certain defects in the international application</p> <p><input type="checkbox"/> Box No. VIII Certain observations on the international application</p>			
Date of submission of the demand  25.05.2005		Date of completion of this report  28.07.2005	
Name and mailing address of the international preliminary examining authority:   European Patent Office - P.B. 5818 Patentlaan 2 NL-2280 HV Rijswijk - Pays Bas Tel. +31 70 340 - 2040 Tx: 31 651 epo nl Fax: +31 70 340 - 3016		Authorized Officer  Bombeke, M  Telephone No. +31 70 340-3576	



**INTERNATIONAL PRELIMINARY REPORT  
ON PATENTABILITY**

International application No.  
PCT/IT2004/000328

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**Box No. I Basis of the report**

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1. With regard to the **language**, this report is based on the international application in the language in which it was filed, unless otherwise indicated under this item.
- ☐ This report is based on translations from the original language into the following language, which is the language of a translation furnished for the purposes of:
- ☐ international search (under Rules 12.3 and 23.1(b))
  - ☐ publication of the international application (under Rule 12.4)
  - ☐ international preliminary examination (under Rules 55.2 and/or 55.3)
2. With regard to the **elements\*** of the international application, this report is based on *(replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to this report)*:

**Description, Pages**

1-3 received on 27.05.2005 with letter of 25.05.2005

**Claims, Numbers**

1-4 received on 27.05.2005 with letter of 25.05.2005

- ☐ a sequence listing and/or any related table(s) - see Supplemental Box Relating to Sequence Listing
3. ☐ The amendments have resulted in the cancellation of:
- ☐ the description, pages
  - ☐ the claims, Nos.
  - ☐ the drawings, sheets/figs
  - ☐ the sequence listing (*specify*):
  - ☐ any table(s) related to sequence listing (*specify*):
4. ☐ This report has been established as if (some of) the amendments annexed to this report and listed below had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).
- ☐ the description, pages
  - ☐ the claims, Nos.
  - ☐ the drawings, sheets/figs
  - ☐ the sequence listing (*specify*):
  - ☐ any table(s) related to sequence listing (*specify*):

\* If item 4 applies, some or all of these sheets may be marked "superseded."

**INTERNATIONAL PRELIMINARY REPORT  
ON PATENTABILITY**

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**Box No. V Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

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**1. Statement**

Novelty (N)	Yes: Claims	1-4
	No: Claims	
Inventive step (IS)	Yes: Claims	1-4
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-4
	No: Claims	

**2. Citations and explanations (Rule 70.7):**

**see separate sheet**

**INTERNATIONAL PRELIMINARY  
REPORT ON PATENTABILITY  
(SEPARATE SHEET)**

International application No.

PCT/IT2004/000328

**Re Item V.**

- 1 The following documents are referred to in this communication:

D1: US 2002/146364 A1 (PRENGAMAN ET AL.) 10 October 2002 (2002-10-10)  
D2: FR 2 420 831 A (ROULEMENTS SOC NOUVELLE) 19 October 1979 (1979-10-19)  
D3: DATABASE WPI Section Ch, Week 200232  
Derwent Publications Ltd., London, GB; Class E36, AN 2002-271433  
XP002303579 & IT 1 304 841 B (CAM SRL) 5 April 2001  
(2001-04-05)  
D4: CN 1 045 957 A (STATE OPERATED NO 752 FACTORY) 10 October 1990  
(1990-10-10)

- 2 The present application meets the criteria of Article 33(1) PCT, because the subject matter of claims 1,2 (method) and claims 3,4 (use) is novel in the sense of Article 33(2) PCT and is determined to involve an inventive step in the sense of Article 33(3)PCT as explained hereinbelow.

- 2.1.1 Document D1, which is considered to represent the most relevant state of the art to the subject matter of claim 1, discloses (par. 005) attrition or ball milling of solid lead particles (derived from lead melt in Barton pot reactor) to produce lead oxide.
- 2.1.2 The subject-matter of independent claim 1 differs from the disclosure of D1 in that lead ingots are reduced in shavings having a helical shape and that said shavings are fed to an abrasion mill (instead of using particulate lead).
- 2.1.3 The problem to be solved by the present invention may therefore be regarded as to find an alternative feed material (rendering the oxidation process in the abrasion mill more efficient), i.e. selecting a more appropriate feed material than coarse pieces, and furthermore to render the known process better controllable

and less cumbersome.

- 2.1.4 D2, the only document which mentions the use of metal shavings/cuttings in the context of powder metallurgy, is directed to the fabrication of specific metal powders. Although abrasion milling of metal shavings and milling of solid metal particles (D2: page 2, lines 4-7) may be regarded as equivalent possibilities for obtaining metal powder, there is no indication or suggestion in D2 to the problem of manufacturing lead particles for batteries nor to the requirement of oxidizing lead particles in a controlled way during abrasion milling thereof. D4 discloses (wet) milling of "granular" lead to be oxidized to PbO. D3 describes an abrasion mill for oxidation milling of Pb particles.
- 2.1.5 From the combined teachings of D2-D4 regarded in the light of D1 the skilled person can hardly derive that helical lead shavings is an obvious alternative and equivalent feed material for producing lead "oxide" powder for battery applications. The solutions proposed in claims 1-4 thus involve the exercise of inventive skill.

## Utilization of lead shavings in abrasion mills for the production of lead oxide

### 1. Technical field

Abrasion mills for the production of lead oxide, like those utilized in the energy storage industry, function through the utilization of lead elements which are  
5 adequately titrated according to the oxidation process for which the mill has been created.

The lead used is produced in titration and ingot - producing pots. The ingots are then transformed into elements of different shapes and dimensions for use in the mills. The shape, size, molecular structure and the other chemical – physical  
10 properties of the lead elements put into the mills, and which make up the "ballast", are fundamental for the good results of the oxidation process, and for the overall cost of the oxide production process.

### 2. Background art

Through the years, the production of material to be used in abrasion mills has  
15 undergone an evolution which has led to several different solutions.

At first, ingots of pure material were used to feed these mills. Although this technique reduces the number of passages to a minimum, it presents numerous disadvantages from the point of view of oxidation process control and yield. Oxidation is a superficial process, and the external surface - volume ratio of the  
20 ingots is low. Moreover, this method does not favor the passage of air through the mill ballast during the oxidation phase, thus reducing the yield of the process.

An important step was made when the ingots were broken down into portions (4, 5 pieces). This intermediate method was an improvement on the first, but maintained the same problems.

25 Many mills utilize a more complex technique with the aim of optimizing the raw materials, although the plant costs considerably more to build and run. The pure lead bars are melted again, molded into cylinders, cooled and matured before being loaded into the mill. This method produces a higher yield of oxide but has created the following serious complications in the system:

- Higher costs for the slug casting machine; higher costs for personnel to run these machines;-
- Higher running costs for safety and management of another lead melting plant;
- Higher costs of conveyor systems for high temperature materials;
- 5 - More space needed for storage areas preceding the mill area;
- Production and management of waste in the slug casting melt pots with a waste of material and problems for its management, re-utilization and elimination;
- Need to plan production in consideration of the great amount of time needed for start up and shut down of the melt pots for the slug casters;

10 **3. Disclosure of invention, best mode for carrying out and industrial applicability**

Abrasion mills for the production of lead oxide call for the use of mechanically produced lead shavings, formed directly from ingots or other adequately titrated lead bars. The **smooth**, helical lead shavings are formed by specially designed

15 machinery and are considerably smaller than the other ~~two~~ types. The mechanical compression and **shear** action carried out by the machine on the cut material produces a **compression and extension stress** which stresses the surface, making it rough and subject to flaking. The mechanical compression favors hardening of the surface of the shaving. These shavings are therefore ready for

20 immediate use inside the mill, with no need for aging.

By virtue of the shape, size and molecular structure, these shavings increase the yield of the oxidation process inside the mill for the following reasons:

- The **smooth** shape increases the external surface – volume ratio, exposing as much material as possible to oxidation and reducing the thermal gradient between

25 the outer and inner areas of each single shaving.

- The helical shape favors the passage of air through the ballast inside the mill, increasing the surface exposed to oxidation and enhancing uniformity of the temperature through the ballast. This second element improves process control and thus increases the quality of the oxide produced, reducing the risk of overheating

30 and the consequent formation of orthorhombic oxide (Massicot).

- The hardening of the shavings' surface increases the abrasion coefficient in the mill ballast, enhancing the heat generated by the abrasion, which is fundamental to the oxidation reaction.

5 - The stressed surface of the shaving tends to flake and release micro-flakes during the rotation of the ballast in the mill, which rapidly offer another surface to the oxidation process.

Another advantage is that abrasion mills fed with shavings for lead oxide production drastically simplify the process system antecedent to the mills and the relative running in the following way:

10 - The mechanical production of the shaving is quick and waste-free, as opposed to fusion processes like slug casters, which produce a great quantity of waste but require *ad hoc* storage, re-use or waste processing systems, due to the particular nature of the material.

15 - The shavings do not need to be stored or aged before using in the mill, thus reducing the volume of the intermediate storage areas and the need to plan and balance production for the line.

- The shavings do not undergo thermal treatment which would change the composition of the previously titrated material, thus assuring the quality of the ballast inside the mill.

20 - Shavings can be produced using simple machinery, low cost and low running costs in terms of number and qualification of the personnel running it.

- The machinery for producing these shavings is utilizable immediately on the basis of the material needed to feed the mill, because they practically do not need long start up or shut down operations.

25 - The use of machinery for producing shavings reduces safety and environmental risks and requirements to a minimum (high temperatures, risk of fire, harmful vapors), increases hourly productivity, in particular as compared to the slug casters used at present, which require the fusion of lead.



# CLAIMS

1. Method of feeding an abrasion mill for the production of lead oxide;  
comprising the steps of:  
5 (a) providing titrated lead in the form of ingots;  
(b) reducing said ingots in shavings having a helical shape; and  
(c) feeding the abrasion mill with said shavings.
2. Method of feeding an abrasion mill according to claim 1, wherein the ingots  
10 are reduced in shavings by cold cutting.
3. Use of titrated lead in the form of shavings having a helical shape for feeding  
an abrasion mill for the production of lead oxide.
- 15 4. Use of titrated lead in the form of shavings according to claim 3, wherein the  
shavings are obtained by cold cutting.